

# **Standard Operating Procedure for the Analysis of PAHs and Atrazine by GC/Ion Trap MS**

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**SOP # CH-IN-003.1**

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## 1.0 Scope And Application

This method is used to determine the concentrations of polycyclic aromatic hydrocarbons (PAHs), atrazine, and degradation products in extracts from vapor, particulate, and precipitation samples. The method is specific for the IADN and LMMB/LMLS projects. The following analytes are measured by this Standard Operation Procedure (SOP).

<u>Analyte</u>	<u>CAS#</u>
atrazine	1912-24-9
desethylatrazine (DEA)	6190-65-4
desisopropylatrazine (DIA)	1007-28-9
acenaphthene	83-32-9
acenaphthylene	208-96-8
anthracene	120-12-7
benzo(a)anthracene	56-55-3
benzo(a)pyrene	50-32-8
benzo(b)fluoranthene	205-99-2
benzo(e)pyrene	192-97-2
benzo(ghi)perylene	191-24-2
benzo(k)fluoranthene	207-08-9
chrysene	218-01-9
coronene	191-07-1
dibenzo(a,h)anthracene	52-70-3
fluoranthene	206-44-0
fluorene	86-73-7
indeno(123cd)pyrene	193-39-5
phenanthrene	85-01-8
pyrene	129-00-0
retene	483-65-8

## 2.0 Summary of Method

This method describes equipment and procedures for operating the GC-Ion trap MS, and instrument optimization specific for PAHs, atrazine and atrazine metabolites. The method is specific for the IADN and LMLS/LMMB atmospheric deposition research projects.

## 2.1 Personnel Restrictions

This method is restricted to use by or under the supervision of an analyst trained and experienced in the operation of gas chromatographs, mass spectrometers, mass spectral and capillary chromatogram interpretation, and data reduction. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.2 Working Linear Range

A multipoint calibration curve will be constructed for each analyte to document the working linear range.

## 2.3 Limit of Detection

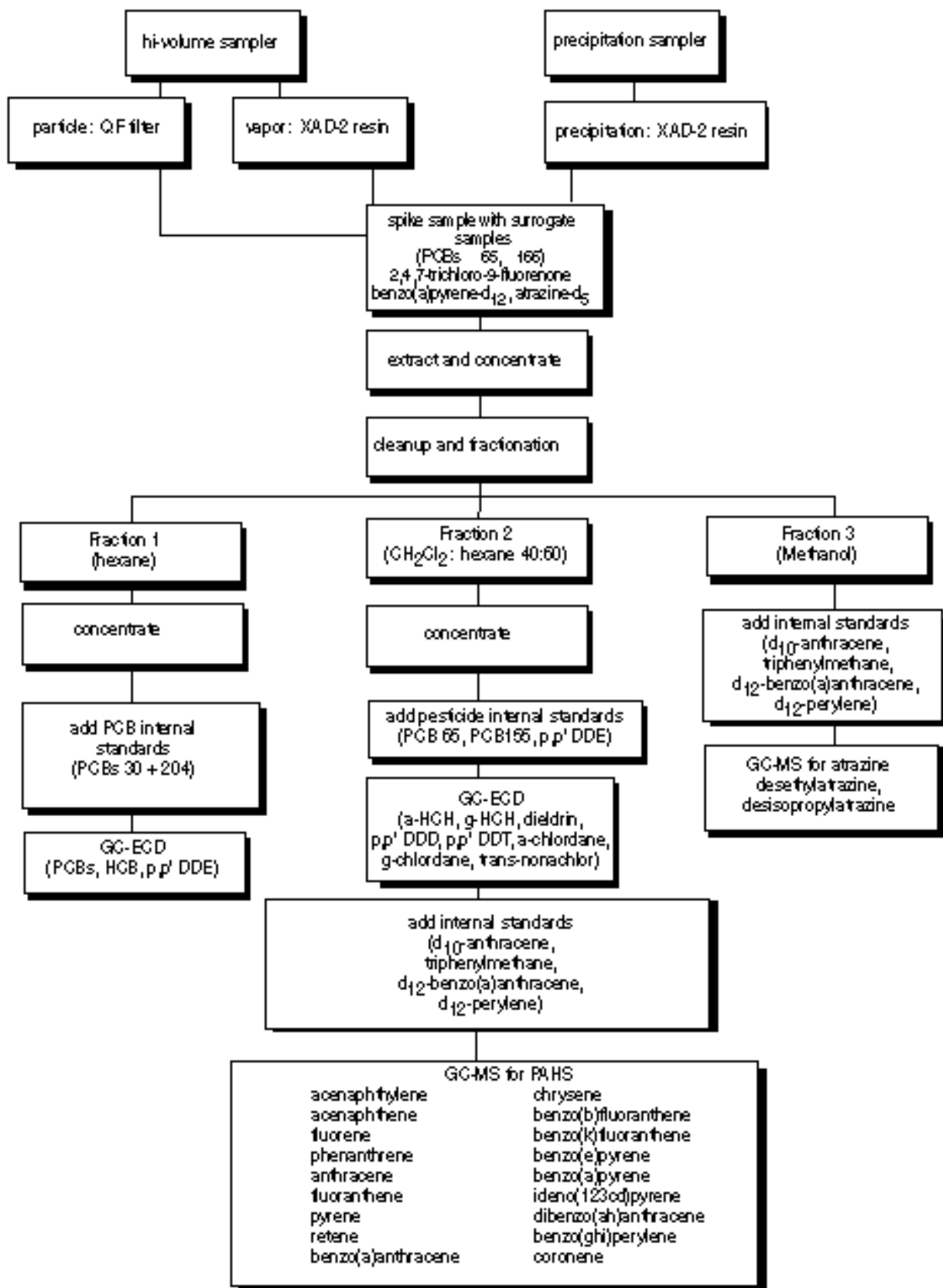
### 2.3.1 IDL

The instrument detection limit (IDL) refers to the smallest signal above background noise that an instrument can reliably detect. The IDL is determined from a data set comprised of three separate chromatographic runs of a low level calibration standard; each run contains 7-10 analyses of the standard. The IDL equals the Student's  $t$  value ( $n-1$ ) multiplied by the standard deviation of this data set.

### 2.3.2 MDL

Method detection limits (MDL) are defined in CFR, Vol 49, No. 209, October 26, 1984, Appendix B to Part 136. Matrix specific MDLs are determined by spiking 7-10 clean matrix samples with the analytes of interest and processing them through the entire extraction, cleanup, and analysis procedure.

## 2.4 Flow Diagram



### **3.0 Safety Precautions**

- 3.1 The toxicity or carcinogenicity of each chemical and reagent used in this method has not been precisely defined. However, each one must be treated as a potential health hazard, and exposure to these chemicals should be minimized. Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin, eyes, etc.
- 3.2 Chemists working in the laboratory should follow ISWS safety rules :
  - 3.2.1 A lab coat is required when working in the lab.
  - 3.2.2 Eye protection with splash resistant safety glasses or safety goggles are required.
  - 3.2.3 Protective gloves should be used while handling samples or standards. Special solvent resistant gloves should be used while handling large amount of solvents.
  - 3.2.4 All solvent work should be done in fume hoods.
  - 3.2.5 Open shoes are not allowed in the laboratory.
  - 3.2.6 Particle mask is required when using dry silica.
  - 3.2.7 Avoid working alone in the laboratory. If work must be performed after hours or in the weekend inform the supervisor or other staff so that your presence is known and will be accounted for in case of an emergency.
  - 3.2.8 Chemicals and solvents are stored under the hoods. Acids must be separated from bases. A rubber bucket is required to transport any chemical.
  - 3.2.9 Gas cylinders should be well secured at all times. Flammable gases are stored in separate storage areas.
  - 3.2.10 Wash hands well after work.
  - 3.2.11 No food or drink is allowed in the laboratory.
  - 3.2.12 In case of minor spillage, get spillage kit to clean the area. A major spill requires the University of Illinois Fire Department to be contacted and the working area evacuated. MSDS sheets are stored in the laboratory and a copy placed on file with the office administrator.
  - 3.2.13 All chemicals and standards must be labeled with chemical name, date, and initials of person to contact.
  - 3.2.14 Empty chemical bottles should be flushed out with water, or, in case of liquid, allowed to evaporate under a hood before discarding.

### 3.3 Waste Disposal

Solvents: Label waste containers, *Chlorinated Waste* and *Non-Chlorinated Waste*. Glass bottles used for waste are placed under hoods for convenience. When full, transfer waste to 10 L carboy containers in solvent cabinet. Contact the ISWS Waste Coordinator for removal.

## 4.0 Apparatus and Materials

### 4.1 Glassware: General Requirements

All glassware, must be meticulously cleaned. Large glassware is thoroughly washed with laboratory detergent and hot water. Glassware with bad stains should be rinsed with MeOH or  $\text{CH}_2\text{Cl}_2$  before using the soap and water procedure. If still not clean, soak in  $\text{H}_2\text{SO}_4:\text{HNO}_3$  (50:50) acid bath overnight, then wash thoroughly with soap and water. Volumetric pipettes used for standards *must* soak in acid bath overnight. Glassware is thoroughly rinsed with tap water, then with DI water and allowed to air dry. The glassware is foil wrapped and heated  $450^\circ\text{C}$  for four hours. If glassware is not clean after muffling at  $450^\circ\text{C}$  for 4 hours, muffle at  $500^\circ\text{C}$  for four hours. The glassware is cooled to ambient temperature and stored in a clean location. Small glassware such as stoppers, vials, and disposables are wrapped in foil or placed into a beaker and covered with foil and heated to  $450^\circ\text{C}$  for four hours, cooled to ambient temperature, and stored in a clean location. Vials are capped as soon as they are removed from the oven. Note: Always use dull side of foil towards glassware. Set initial temperature of furnace to  $200^\circ\text{C}$  if possible.

### 4.2 Vortex Mixer

### 4.3 Volumetric flasks and pipets - Class A, various sizes

### 4.4 Auto sample vials, amber, 2 mL with 200 $\mu\text{L}$ inserts, caps and teflon-lined septa

### 4.5 Positive displacement micro pipet, glass capillaries (Drummond or equivalent)

### 4.6 GC-Ion Trap MS (see Section 9.0 for detailed information)

4.6.1 Model: Varian Saturn 3, capillary, GC/Ion trap-MS system

4.6.2 Injector: SPI, 8200 autosampler, LB2 Thermogreen septa (Supelco)

4.6.3 Column: 30 m x 0.25  $\mu\text{m}$  x 0.25 mm, DB-5 MS (J & W Scientific)

### 4.7 Chemicals/Standards

Pesticide residue quality or equivalent. All reagents are evaluated for interferences using laboratory blanks.

#### 4.7.1 Solvents, EM Omnisolve or equivalent

##### 4.7.1.1 Hexane

4.7.1.2 Methanol

4.7.1.3 Acetone

4.7.2 Standards

4.7.2.1 Atrazine and PAH Stock Standards

Stock standard solutions are purchased from commercial sources (Ultra Scientific, Accustandard, Chem Service, Crescent Chemical) or are obtained from the USEPA repository. When stock solutions are not available, pesticides are purchased as the neat material and gravimetrically prepared in house.

4.7.2.2 Surrogate Standard Solutions

4.7.2.2.1 Surrogate standards are purchased from commercial sources (Aldrich Chemical, Cambridge Isotope) or are obtained from the USEPA repository. The following surrogate standards are utilized:

atrazine-d5 (atrazine surrogate)  
benzo-(a)pyrene-d12 (PAH surrogate)

4.7.2.2.2 If stock solutions are not commercially available, they are gravimetrically prepared from the neat material. Individual stock solutions are serially diluted in volumetric flasks to obtain the surrogate spike standard/s. A combined surrogate spiking standard may be prepared to save sample preparation time during the extraction procedure.

4.7.2.2.3 All samples are spiked with surrogate standards prior to extraction using volumetric pipets or a Drummond pipet and the spike volumes recorded on the sample preparation log.

4.7.2.3 Internal Standard Solutions (ISTDs)

4.7.2.3.1 Internal Standards are purchased commercially (Ultra Scientific) as a stock standard or as the neat material. The following ISTDs are utilized:

anthracene-d10 (PAH and atrazine ISTD)  
benzo(a)anthracene-d12 (PAH ISTD)  
perylene-d12 (PAH ISTD)  
triphenylmethane (PAH ISTD)

4.7.2.3.2 If stock solutions are not commercially available they are gravimetrically prepared from the neat material. Individual stock



solutions are serially diluted in volumetric flasks to obtain the ISTD spiking standard/s.

- 4.7.2.3.3 ISTDs are added to the appropriate sample fraction (PAHs in 40% DCM, and atrazine in MeOH) prior to GC-MS analysis. A Drummond micropipet is used for ISTD addition.

#### 4.7.2.4 Chromatographic Calibration Standards

Combined instrument calibration standards are prepared from the individual stock standards by volumetric dilution to obtain five concentration levels. The calibration standard concentrations bracket the expected analyte amounts in samples assayed and are within the working linear range of the detectors. Calibration mixes are prepared specifically for the appropriate instrument and fraction analyzed. The following calibration mixes are prepared.

PAHs with surrogate and ISTDs  
atrazine, DEA, and DIA with surrogate and STDs.

#### 4.7.2.5 Matrix Spiking Solutions

- 4.7.2.5.1 Combined matrix spiking solutions are prepared from the individual stock standards by volumetric dilution. Combined matrix spike solutions are prepared for each analyte group. The following matrix spike mixes are prepared:

PAHs  
atrazine, DEA, DIA.

- 4.7.2.5.2 The matrix spike solutions will be added to clean sample matrix material prior to extraction to calculate the recovery of individual analytes. One matrix spike will be extracted with each batch of samples. The matrix spike will be added to the sample using a Drummond micropipet or a volumetric pipet and the spiking amounts reported in the sample preparation log. Detailed sample preparation procedures can be found in SOP #CH-PR-001.3, March 1995.

#### 4.7.2.6 Standard Evaluation

New working standards will be assayed prior to use by comparison with existing standards. Standards must agree within 10% prior to use.

### 4.7.3 Gases

- 4.7.3.1 Helium, carrier gas, 99.9999% chromatographic grade

- 4.7.3.2 Carbon Dioxide, SPI coolant, general grade

4.7.3.3 Air, autosampler pressurizing gas, general grade

## 5.0 GC/MS System Evaluation

Prior to each run the GC/IT-MS system performance and calibration are verified for all analytes. A mass spectrometer tune is performed prior to each run using system software with perfluorotributylamine (PFTBA) calibration gas. Adjustments or maintenance is performed such that selected calibration masses and their respective isotopes meet the target mass-intensity criteria.

Hexane is injected immediately prior to each run to ensure the system is free of contaminants or interfering peaks.

Records of daily system performance and maintenance are maintained.

***Note:** The system is evaluated and tuned with the column and injector set at the highest temperature attained during a normal acquisition (i.e., column at 300°C and injector at 289°C). This is to assure optimal conditions for the high-boiling compounds (such as coronene).*

### 5.1 Daily Evaluation

#### 5.1.1 Check air/water for leaks.

Set mass range to 10-45 m/z; insure AGC is off; set ion time to 300  $\mu$ sec; set filament emission current to 10  $\mu$ A; turn on the filament, multiplier, and RF; normalize the peaks. The following conditions indicate there is no evidence of a significant air leak or water background present:

- a) There are discrete peaks at 18, 28, and 32.
- b) The peak at 28 is higher than that at 18 and the 28:32 ratio is about 4:1.
- c) The ratio of peak 18 to peak 19 is 10:1 or greater.
- d) The 100% counts value is less than 500.
- e) The TIC value is less than 2000.

#### 5.1.2 Check PFTBA calibration gas level.

The ionization time should read between 500-1000  $\mu$ sec.

#### 5.1.3 Check the valley to isotope % of ions 131 and 132.

The value should be around 25%. This means the 132 isotope peak is four times the height of the valley between ions 131 and 132. Adjust tune parameters, if necessary, to obtain the proper valley/isotope %:

- a) Begin by setting the mass range to 127-137 m/z. Turn on the multiplier, filament, RF, calibration gas, and AGC. Set the AGC target value to 20000.
- b) Adjust the A/M (axial modulation) voltage until the valley/isotope % appears to be close to 25%. The axial modulation value is typically between 2.5 and 5 volts.

- c) Run the *Set AGC Target* program. The AGC target should be optimally set to 20000 - 25000. If not, the A/M voltage and/or the electron multiplier voltage may need to be (re)adjusted.
- 5.1.4 Run a mass calibration with PFTBA calibration gas. Verify valid calibration prior to proceeding.
- 5.2 Weekly Evaluation
- 5.2.1 Check integrator zero.  
When both the RF and the multiplier are on, the measured signal should be between 0.2 and 0.8 ADC counts. If the integrator zero is not within these limits, it must be adjusted.
- 5.2.2 Set AGC target.  
The *Set AGC Target* program should be run to verify the valley/isotope % is actually 25%. This program is also a check on the electron multiplier value. The optimal target value is 20000.
- 5.3 Monthly Evaluation
- 5.3.1 Check multiplier voltage.
- 5.3.2 Check filament emission current.  
The filament emission current is set to 10  $\mu$ A. The *Set Filament Emission Current* program should be ran to verify instrument performance. If the program wants to set the filament emission current higher, this is usually indicative of a high level of background.
- 5.3.3 Check RF voltage ramp  
The RF voltage ramp should rise gradually in a generally straight line from low mass to high mass throughout the entire mass range, without any sudden rises in the ramp. The *average* response value should be 300 - 500 ADC counts and the *highest* response value should be 500-900 ADC counts.
- 5.3.4 Check carrier gas flow rate.  
The optimum carrier gas volumetric flow rate into the ion trap is 1 mL/min. at the maximum temperature reached during an analysis. This is determined by measuring the time required for an unretained compound, such as air, to elute from the column, and then calculating the volumetric flow rate using the following formula:

$$\text{Volumetric flow rate (mL/min)} = \frac{\delta r^2 l}{t}$$

Where:

$\delta$  is 3.14

$r$  is the radius of the column (cm)

$l$  is the length of the column (cm)

$t$  is the retention time of the unretained compound (min)

## **6.0 Periodic GC/MS Maintenance**

- 6.1 Change injector septa after approximately 100 injections.
- 6.2 Vent rough pump a minimum of two times per month.
- 6.3 Change hexane in injector rinse reservoir every month.
- 6.4 Change waste arm septa every month.
- 6.5 Change rough pump oil every three to six months.

## **7.0 Preparation of Autosampler Vials for GC/MS Analysis**

- 7.1 Remove standards and samples from freezer; equilibrate to ambient temperature (approximately two hours).
- 7.2 Initiate sample log sheet for GC/MS run. Each analytical run consists of a hexane blank, standards to establish a calibration curve, a performance check standard if available, samples, a calibration check standard every five to seven samples, and a final calibration check and hexane blank.
- 7.3 Label autosampler vials for samples, standards, and hexane blanks.
- 7.4 Insert 200  $\mu$ L glass insert into each vial (except in cases of sample dilutions).
- 7.5 Spike samples with ISTD if necessary. (See Section 8.0 for spiking procedure.)
- 7.6 Before transferring samples and standards to the corresponding labeled autosampler vials, mix contents of each vial and bottle well by holding on a vortex mixer for 5-10 seconds. Use muffled Pasteur pipettes for transfer. The glass insert should be filled approximately half full.
- 7.7 Place cap onto autosampler vial after it is filled.
- 7.8 Load autosampler vials onto autosampler.

## **8.0 Spiking Samples with ISTD**

- 8.1 Remove ISTDs from freezer; equilibrate to ambient temperature (approximately two hours). Vortex ISTD solution to mix.
- 8.2 Clean micropipette:

Remove glass tube used to cover plunger. Rinse plunger with  $\text{CH}_2\text{Cl}_2$ . Allow to dry. Without manually touching glass tubes, insert plunger into a new glass tube. Tighten tube in position.

Rinse pipette tube:

- 1) Draw up  $\text{CH}_2\text{Cl}_2$  into pipette and discard into solvent waste container.
- 2) Draw up hexane into pipette and discard into solvent waste container. Repeat five times.  
Allow to dry.
- 3) Draw up internal standard into pipette and discard into solvent waste container. Repeat two times.
- 4) Fill micropipette.

8.3 Spike sample vial. See chart below for internal standard and amount.

Fraction	Compound	Type of Sample	Internal Standard	Spike Volume (μL)	Final Mass in Sample (ng)*	Color of Dot on Label
Hexane	PCBs and Pesticides	vapor, particle, and rain	PCB 30	100	8	Red
			PCB 204		6	
40%	Pesticides	vapor, particle, and rain	PCB 65	100	23.7	Blue
			PCB 155		17.5	
			DDE		20	
40%	PAHs	vapor, particle, and rain	d10-Anthracene	100**	200	Black
			Triphenylmethane		100	
			d12-Benzo(a)anthracene		200	
			d12-Perylene		200	
MeOH	Atrazine	vapor, particle, and rain	d10-Anthracene	100	200	Black

\*Values in this column are approximate and may change slightly depending on the exact concentrations of the compounds in the stock solutions.

\*\*If the sample is expected to be high in concentration of PAHs or if the final sample volume is greater than 2 mL, more ISTD solution may be added in increments of 100μL.

8.4 Mark each sample vial label with the appropriate color of dot to verify sample has been spiked. (Use water-proof marker.)

8.5 Rinse with solvent and replace glass tube used to cover plunger of micropipette. Store micropipette.

8.6 Mark sample box with the following information using the same color of pens as dots on vial labels:

Date sample vials spiked.  
Fraction spiked.  
Initials of chemist spiking.

## 9.0 Normal Operating Parameters for Saturn 3 GC/MS

9.1 Operating software: Version 5.0 or later

9.2 Operating mode: EI (electron impact)

- 9.3 AGC (automatic gain control): ON
- 9.4 AGC prescan factors:
- |                  |    |
|------------------|----|
| Background mass: | 98 |
| Data steps:      | 50 |
| Weight factor:   | 1  |
- 9.5 Scan rate variables:
- |                   |      |
|-------------------|------|
| Scan time (msec): | 1000 |
| Micro-scans:      | 15   |
- 9.6 EI/AGC parameters:
- |                                     |       |
|-------------------------------------|-------|
| EI background mass (m/z):           | 98    |
| EI maximum ionization time (µsec):  | 25000 |
| AGC prescan ionization time (µsec): | 100   |
| AGC prescan storage level (m/z):    | 20.0  |
| Data steps in AGC prescan:          | 50    |
| RF dump value (m/z):                | 650.0 |
| AGC weight factor:                  | 1     |
- 9.7 Scan segment breaks:
- | <u>Seg masses</u>  | <u>Seg RF</u> | <u>Seg time</u> |
|--------------------|---------------|-----------------|
| Segment 1: 10-98   | 10            | 100             |
| Segment 2: 99-310  | 10            | 100             |
| Segment 3: 311-399 | 10            | 100             |
| Segment 4: 400-650 | 10            | 100             |
- 9.8 PAH Operating Conditions
- 9.8.1 *SPI Injector*
- |                      |   |
|----------------------|---|
| Injection volume:    | 1 µL                                    |
| Hot needle time:     | 0.03 minutes                            |
| Injection time:      | 0.01 minutes                            |
| Injection rate:      | 0.5 µL/second                           |
| Lower air gap        | yes                                     |
| Upper air gap        | yes                                     |
| Needle depth         | 90%                                     |
| Initial temperature: | 50°C, hold 0.10 minutes                 |
| Ramp 1:              | 200°C/min. to 290°C, hold 48.70 minutes |
- 9.8.2 The GC temperature program is optimized to achieve >50% resolution for all analyte peaks and any known interferant. Typical conditions are as follows:
- |                      |                        |
|----------------------|------------------------|
| <i>Oven Program:</i> | 50 minutes             |
| Initial temperature: | 75°C, hold 2.0 minutes |
| Ramp 1:              | 25°C/min. to 150°C     |
| Ramp 2:              | 4°C/min. to 235°C      |

Ramp 3:	3 °C/min. to 265 °C
Ramp 4:	50 °C/min. to 300 °C, hold 11.94 minutes

#### 9.8.3 Miscellaneous Parameters

Column head pressure:	15 psi
Transfer line:	300 °C
Column linear velocity:	1.0 mL/min. at 300 °C
Septum purge flow:	4.25 mL/min.
Manifold set temperature:	240 °C
Mass range:	98-310 m/z
Mass defect:	50 mu/100μ

#### 9.8.4 Calibration Information

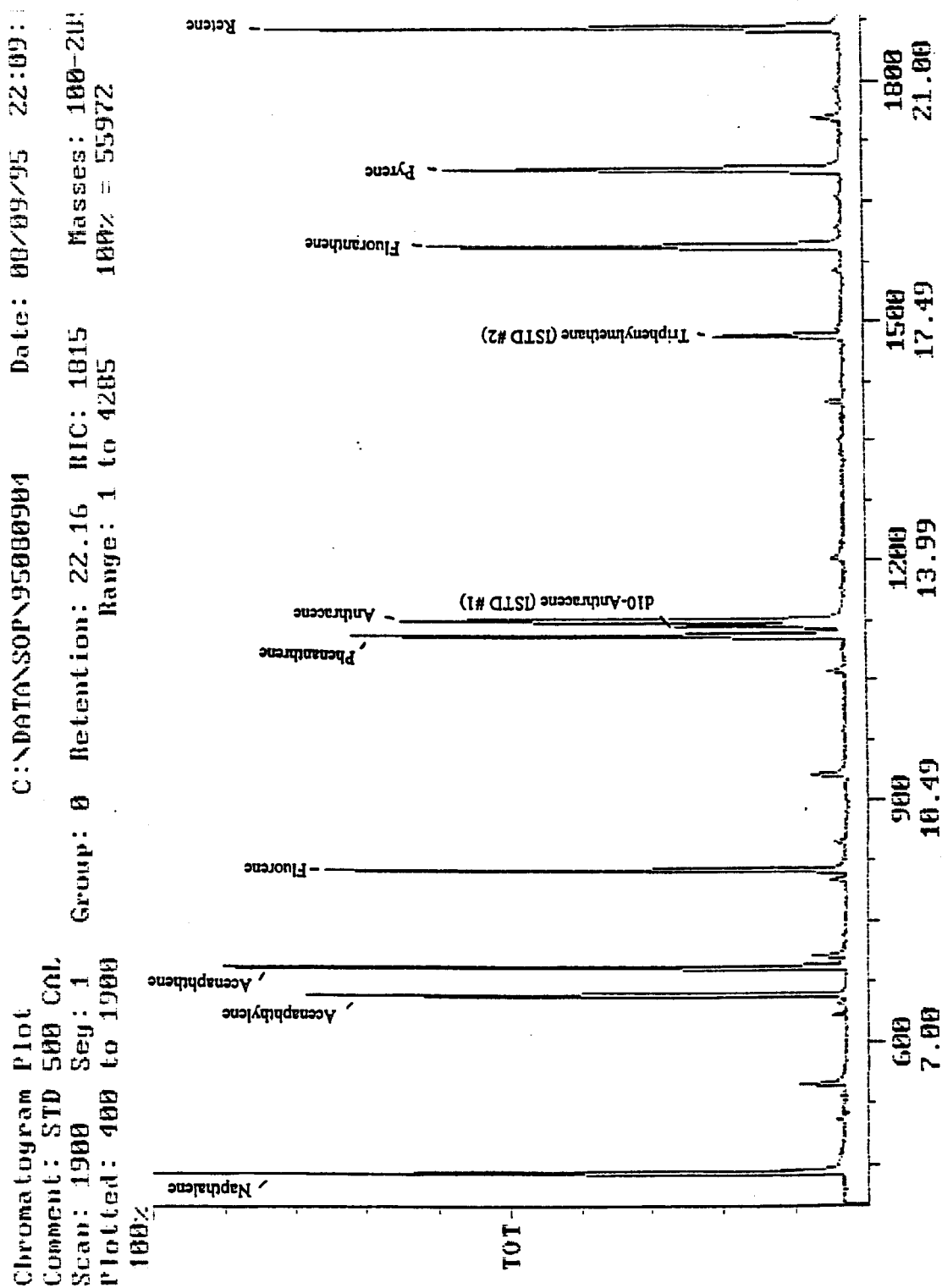
A multipoint calibration curve is prepared for each analyte prior to each run. A calibration check is performed every 5-10 samples and at the end of each run. Recalibration is required if a shift of >20% is observed. The method of internal standard calibration is utilized with four internal standards for PAHs and one for atrazine. The chromatogram is divided into four time segments with each segment calibrated relative to one internal standard response for the PAH runs.

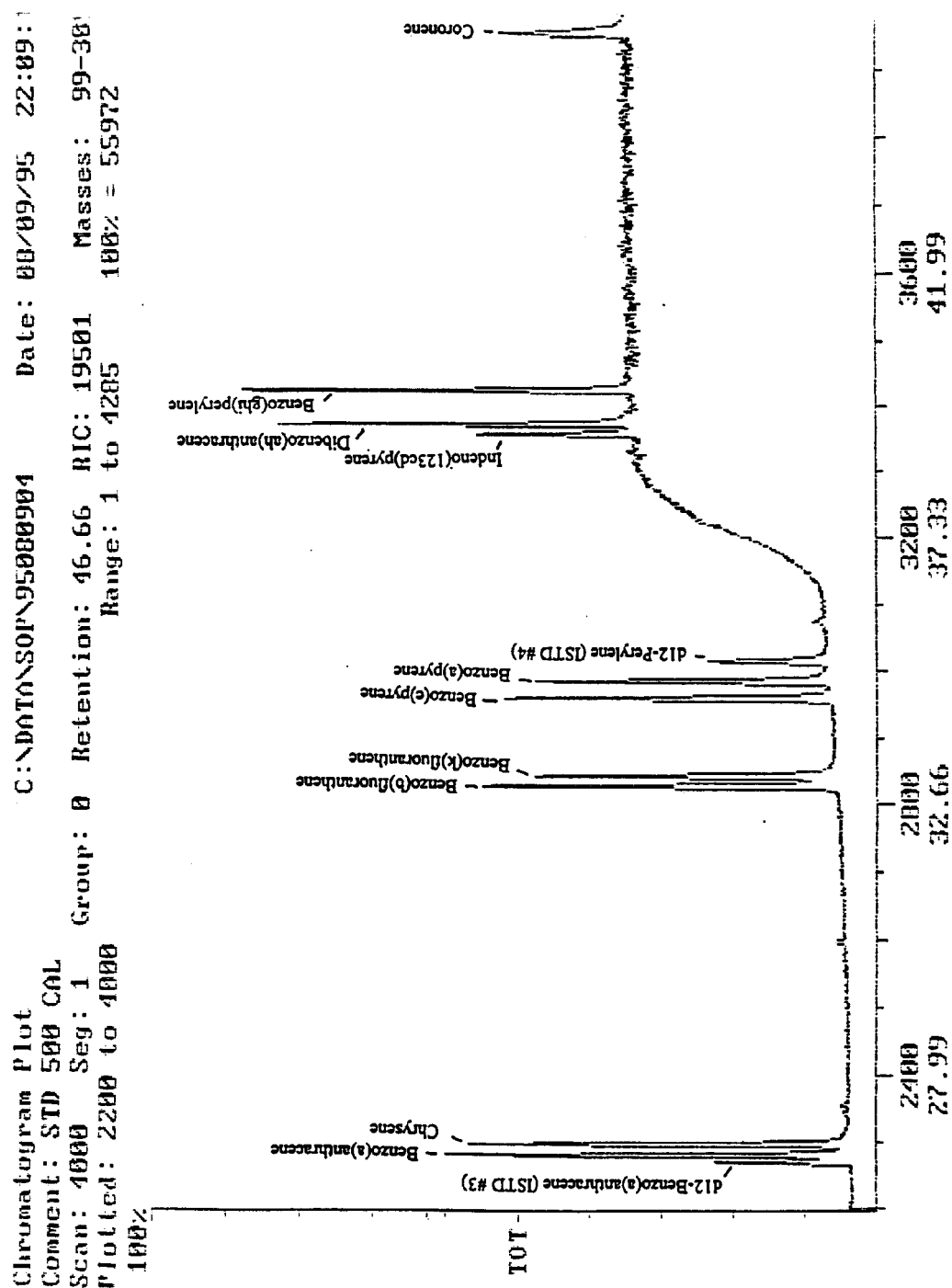
Data is collected in the full-scan mode, however, selected ions are used for quantitation.



Retention Time	ISTD #	Name of Compound	Quantitating Masses
8.03	1	Acenaphthylene	152
8.46	1	Acenaphthene	153
9.95	1	Fluorene	165+166
13.54	1	Phenanthrene	178
13.68	1	d10-Anthracene (ISTD #1)	188
13.76	1	Anthracene	178
18.02	2	Triphenylmethane (ISTD #2)	165+244
19.36	2	Fluoranthene	202
20.5	2	Pyrene	202
22.58	3	Retene	219+234
27.38	3	d12-Benzo(a)anthracene (ISTD #3)	240
27.5	3	Benzo(a)anthracene	228
27.7	3	Chrysene	228
34.25	4	Benzo(b)fluoranthene	252
34.44	4	Benzo(k)fluoranthene	252
35.89	4	Benzo(e)pyrene	252
36.09	4	d12-Benzo(a)pyrene (surrogate)	264
36.21	4	Benzo(a)pyrene	252
36.59	4	d12-Perylene (ISTD #4)	130+132+264
40.68	4	Indeno(123cd)pyrene	138+276+277
40.87	4	Dibenzo(ah)anthracene	139+278+279
41.56	4	Benzo(ghi)perylene	138+276+277
48.51	4	Coronene	150+300+301

9.8.5 PAH chromatogram- full scan mode





9.9 Atrazine Operating Conditions

9.9.1 SPI Injector

Injection volume: 2 µL  
Hot needle time: 0.03 minutes  
Injection time: 0.10 minutes  
Injection rate: 0.2 µL/second  
Lower air gap yes  
Upper air gap yes  
Needle depth 90%  
Initial temperature: 50°C, hold 0.85 minutes  
Ramp 1: 200°C/min. to 290°C, hold 29.95 minutes

9.9.2 Oven Program: 32 minutes

Initial temperature: 75°C, hold 2.0 minutes  
Ramp 1: 25°C/min. to 150°C  
Ramp 2: 4°C/min. to 190°C  
Ramp 3: 50°C/min. to 300°C, hold 14.80 minutes

9.9.3 Miscellaneous Parameters

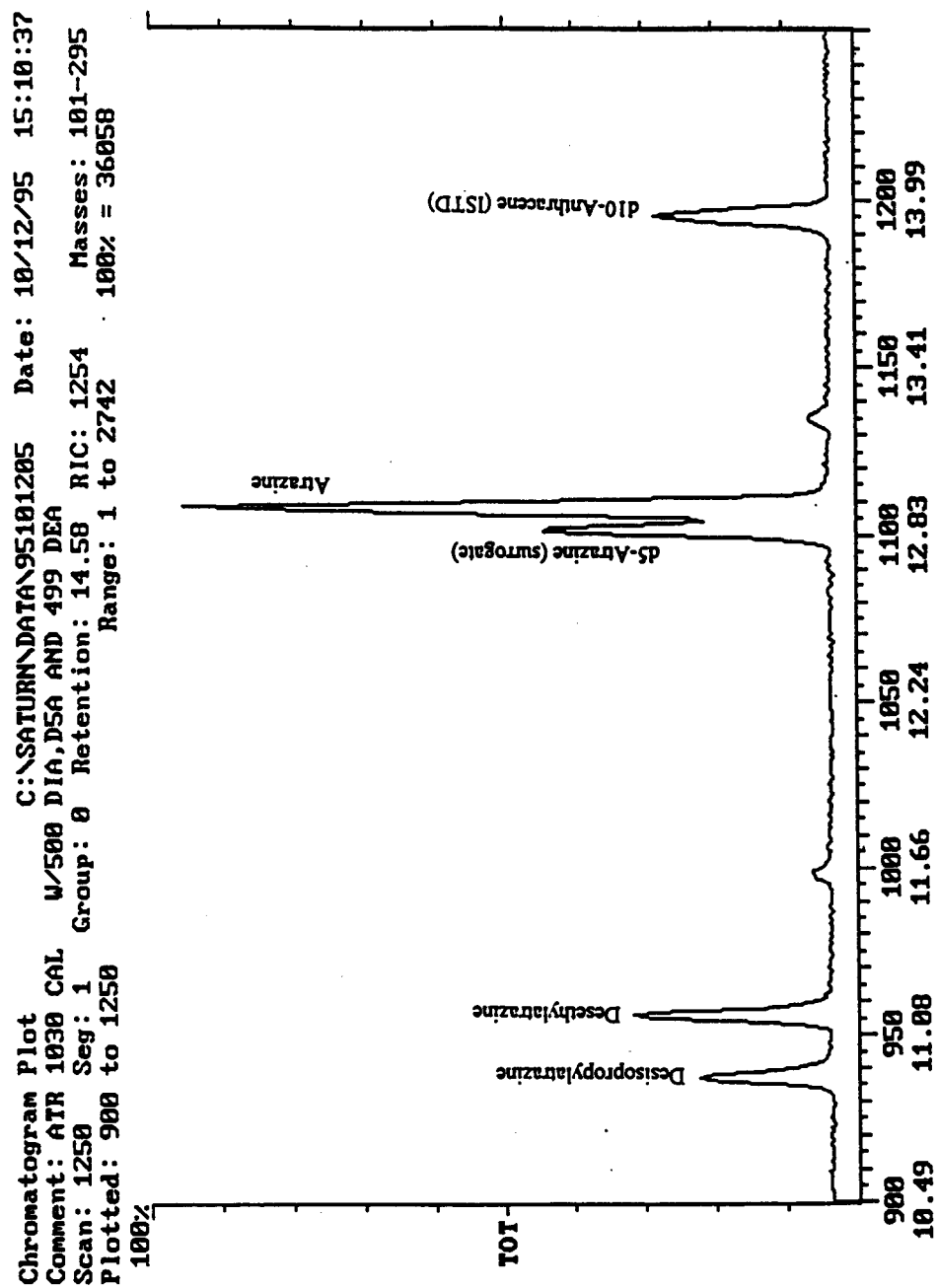
Column head pressure: 15 psi  
Transfer line: 300°C  
Column linear velocity: 1.0 mL/min. at 300°C  
Septum purge flow: 4.25 mL/min.  
Manifold set temperature: 240°C  
Mass range: 98-310 m/z  
Mass defect: 50 mu/100µ

9.9.4 Calibration

See Section 9.8.4 for a general discussion of the calibration procedure.

Retention Time	Name of Compound	Quantitating Masses
10.73	Desisopropylatrazine	158+173
10.93	Desethylatrazine	172
12.61	d5-Atrazine (surrogate)	205+220
12.69	Atrazine	200+215
13.64	d10-Anthracene (ISTD)	188

## 9.9.5 Atrazine chromatogram - full scan mode



## 10.0 Data Evaluation

- 10.1 View the hexane blank total ion chromatogram to determine if the system is clean.
- 10.2 Integrate each standard using the previous calibration curve. Print a hard copy of each standard data report.
- 10.3 Build a new calibration curve daily or for each sample set using a minimum of three standards.
- 10.4 Evaluate the performance check standard (US 106) for PAHs. Print a hard copy of the data report.
- 10.5 Integrate each sample, peak observing each individual compound. Print a hard copy of compounds that are manually integrated and a hard copy of the final sample report.
- 10.6 Continue evaluating each sample, each calibration check (every five to seven samples), and the final hexane blank chromatogram.
- 10.7 Print a hard copy of the tune conditions for the run.
- 10.8 After the entire run has been integrated and evaluated, run the macro *Print.prc* (see Appendix A) to convert Saturn data files to a format for import into Quattro® Pro.
- 10.9 Import the Saturn data to a Quattro® Pro worksheet.
- 10.10 Check the data in the worksheet against the Saturn hard copies. Correct the worksheet and add extraction batch codes and any necessary comments.
- 10.11 Print out a hard copy of the Quattro® Pro worksheet and compare the data again to the Saturn hard copies. Initialize the folder containing the Saturn hard copies as being checked.
- 10.12 Copy the worksheet data into the appropriate spreadsheet.
- 10.13 Send the Saturn hard copies and a disk copy of the updated Quattro® Pro spreadsheet to the lab supervisor for final review.
- 10.14 Saturn data files must be backed up before they are deleted from the Saturn system hard drive. Make two backups using optical disks or magnetic tapes.
- 10.15 The data will be considered valid if the calibration check standards and the performance check standard are within 20%.
- 10.16 Formula for manual-calculations of GC/MS Data

Some over-range peaks may require manual calculations to determine the analyte concentration

$$Mass_{smp} = Area_{smp} \times \left\{ \frac{Mass}{Area} \right\}_{cal} \times \left\{ \frac{Area}{Mass} \right\}_{cal} \times \left\{ \frac{Mass}{Area} \right\}_{smp}$$



## Appendix A. Macros

### PRINT.PRC MACRO

```
screen 1:
\ PRINT
# $pathname = "D:\DATA\"
# $areafile = "B:\AR"
# $amntfile = "B:\AM"

screen 2:
\ PRINT
CLS
ROW 10
COLUMN 10
PRINT "Enter subdirectory: "
INPUT-STRING
CHOP-TRAILING-BLANKS-FROM $STRING
JOIN-STRINGS $pathname $STRING
JOIN-STRINGS $pathname "\"
JOIN-STRINGS $areafile $STRING
JOIN-STRINGS $areafile ".PRN"
JOIN-STRINGS $amntfile $STRING
JOIN-STRINGS $amntfile ".PRN"

screen 3:
\ PRINT
CLS
ROW-COL 3 15
FILE-LIST-OF "*.QD"
$FILE-LIST-PATH = $pathname
FILE-LIST-TITLE "Select Quant. files"
FILE-LIST-SIZE = 10
SHOW-FILE-LIST

screen 4:
\ PRINT
CREATE-FILE $areafile
WRITE-TO-FILE
FOR J = 1 TO #FILES-IN-LIST
USE-DATA-FILE $FILE-LIST-NAME# J
PRINT 1 "
PRINT $DATA-SAMPLE-ID
PRINT 1 "
PRINT 1 ,
PRINT 1 "
PRINT $DATA-FILE-NAME
```





## **Appendix A. Macros (Cont'd)**

```
PRINT 1 "  
PRINT 1 ,  
PRINT 1 "  
PRINT-DATE(1)-OF DATA-FILE-DATE  
PRINT 1"  
PRINT " , ,"  
USE-QUAN-FILE $FILE-LIST-NAME# J
```

```
screen 5:  
\ PRINT  
FOR I = 1 TO #QUAN-COMPOUNDS  
READ-QUAN-COMPOUND# I  
FIELD L7.0 PRINT QUAN-PEAK-AREA  
PRINT 1 ,  
NEXT  
CR  
NEXT  
CLOSE-FILE
```